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J. R. Fritze

Development of a Method for the
Microscopic Study of Metals at elevated
Ranges of Temperature

DEVELOPMENT OF A METHOD FOR THE
MICROSCOPIC STUDY OF METALS AT ELEVATED
RANGES OF TEMPERATURE

BY

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THESIS

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

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.....of Metals at Elevated Ranges of Temperature.....

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF.....Bachelor of Science in Chemical Engineering.....

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PART I

DEVELOPMENT OF A METHOD FOR THE
MICROSCOPIC STUDY OF METALS AT ELEVATED
RANGES OF TEMPERATUREI INTRODUCTION

1. Purpose of the present investigation.

There were three reasons for undertaking this work. First; to construct a simple apparatus and develop a method for the microstudy of metals at elevated ranges of temperature. Second: To study more closely the relation between the changes in structure and the different thermal critical points and if possible to actually see these changes as they occurred. Third: to study the effect of so called "neutral" gases on steel at these elevated temperatures. A neutral gas can be defined as any gas that will not cause a chemical change to take place on the surface of the steel. No attempt was to be made to use a vacuum because this would involve a much more complex apparatus than was desirable.

2. The present status of the field.

Up to the present time, the attempts to study microscopically the structural changes in steel as these changes are actually taking place, have been very few. However, several unique attempts to arrive at this end are worth mentioning.

The first is that of J. G. Ayers, Jr.(1) He took a steel bar and heated one end of it to well above its critical range

(1) Proceedings of the American Society for Testing Materials.
Part II 1918 page 88.

by allowing this end only in the heat and allowing the other end to remain outside the furnace and well below any critical temperature. The steel upon quenching contained at some point a structure similar to any separately heat treated samples and showed from one end to the other of the bar a continuous record of transformation.

A piece of normalized .50% carbon steel 100 mm long and 12 mm in diameter, milled off to obtain a 7 mm face for its full length, was chosen and treated as described above. The specimen was quenched all over in cold water and 1 mm was then ground off to remove any possible effect of surface decarburization. This grinding was done with extreme care in order to prevent any tempering effect on the steel. The specimen was then polished and etched in a 5% solution of picric acid in alcohol and sixteen overlapping photomicrographs of 100 diameters were taken from a point in the specimen entirely unaffected by any critical change to a point almost above the maximum effect of the critical transformation. These sixteen pictures were then matched together and mounted as one photograph. From it, the successive changes are very easily followed.

Another attempt is that of P. Oberhoffer, a German chemist.(2) He developed an apparatus by means ^{OF} which metals may be microphotographically examined while they are heated to high temperature in vacuo. The author heated the specimen until a change was in progress, stopped the change by instant cooling and then photographed it; he then reheated to restart the change and after an

(2) Zeitschrift fur Electrochemie V. 15 1919 page 634.

Abstracts: Chem. Abstracts 1910 page 160.

Journal of the Society of Chemical Industry V. 28 1909
page 1042.

interval recooled and again photographed the stage to which the change had arrived and so on to its completion.

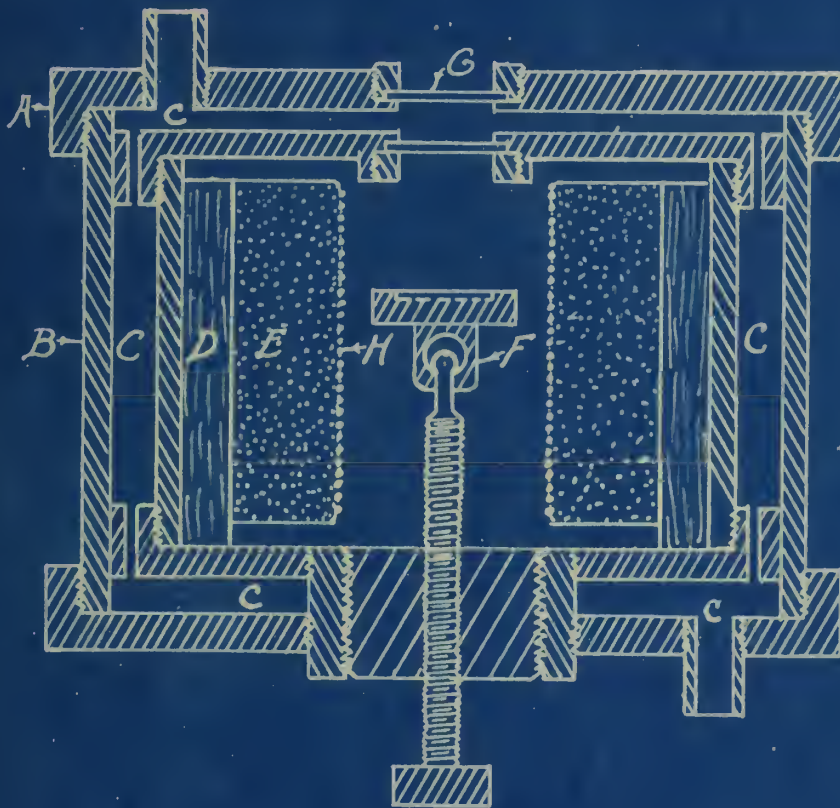
Minute quantities of gases were introduced into the vacuum and the behavior of the polished specimen under their influence was examined. Hydrogen and chlorine were both utilized. The apparatus used consisted of a small and compact furnace surrounding the section for examination and designed to rest upon the stage of a Le Chatilier microscope. The innermost shell of the furnace was of glass in which arms were blown for the introduction of thermo couple wires, platinum wire for heating and vacuum connection. This was surrounded by a thin covering of copper containing an aperture at the bottom to permit observation through the glass. The whole was surrounded by a^d water jacket. A quartz tube wound with platinum wire constituted the heating element. A thin sheet of platinum with aperture served to protect the glass bottom of the apparatus from heat radiated by the hot section. The author studied with this apparatus the transformation of austenite to sorbite, but although his results were very promising, no definite statements could be made.

II APPARATUS USED.

1. Construction of Furnace.

The furnace, Plate 1, was constructed in two parts and arranged so that the heating element could be taken out without injuring the outer casing. The walls of the jacket part were made of thin brass tubes and the tops and bottoms were made of solid brass disks molded especially for this work. The inner and outer tubes of the furnace were held in place by a threaded brass bushing and reinforced by a coating of solder. In the center of each of the brass caps there was a thin sheet of mica held in place by a thread-

*Cross Section of Furnace used in Heating
the Speciman (Actual Size)*



*A Brass Cap
B Brass Tube
C Water Space
D Alundum Tube*

*E Alundum Cement
F Brass Screw & Cap
G Mica Window
H Heating Coil*

THE
OF THE
CAPACITY OF ALUMINUM



Figure 1. Set up of apparatus.

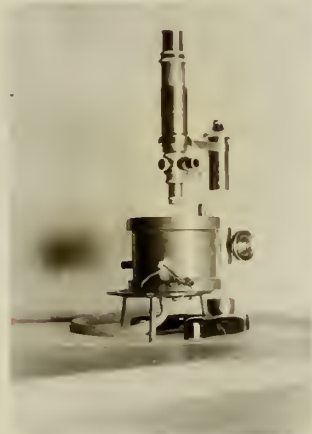


Figure 2.

Microscope and Furnace.

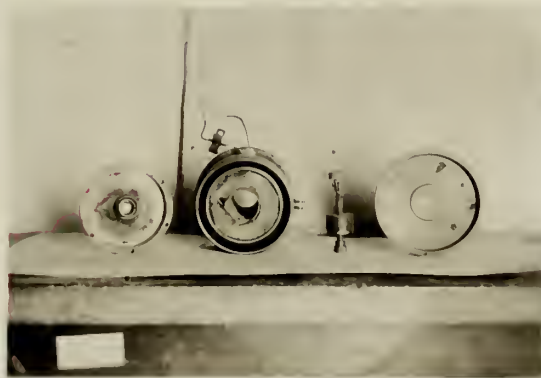


Figure 3. Furnace dismantled.

ed bushing which screwed down into a seat in the cap itself and was made water tight by means of asbestos washers. The specimen holder consisted of a brass plug, which fitted into the bushing supporting the cooling jacket, and a threaded screw terminating in a universal joint at its top. The plug and screw could be removed from the furnace, specimen put on, thermocouple inserted through a hole in the plug, and both then returned to their former position. Fitting snugly inside of the inner brass was a thick walled, porous alundum tube which served as a support for the cement and a heat insulator for the cooling jacket. Especial care was taken to obtain a heating coil that would heat uniformly. No. 18 nichrome wire was wound around a wooden core one and one eighth inches in diameter. A strand of asbestos string was wound on the coil in between each turn of the wire so that good insulation and equal spacing was obtained throughout. The coil and core were then put in the proper place in the furnace and packed with alundum cement. This cement was dried by passing through the coil, two amperes for one hour. The current was then gradually increased until twelve amperes were reached. After one hour at this temperature the wooden core had charred to such an extent that it was easily removed. In putting on the caps, the top of the tubes were first coated with a mixture of graphite and oil, thus forming a water tight joint when tightly screwed down. The furnace was then ready for use.

(2) Microscope.

The microscope used was manufactured by E. Leitz of Wetzlar, Germany. The stage was removed but no other changes were necessary for this work. The furnace was placed on a tripod that had been cut down to about two inches in height. With the movable

stage gone, the microscope then fitted very nicely over the furnace. A Leitz achromatic objective with a focal length of 32mm and an initial magnification of 3.2 diameters was used. Such a large focal length was necessary in order to permit focussing when the specimen was well down in the furnace. This was used in connection^{with} a Leitz No. 5 Huygenian eyepiece with focal length of 20.85 mm and an initial magnification of 12 diameters, this combination giving a magnification at the eyepiece of 38 diameters. A plain disk illuminator was used. A reflecting prism was also tried but did not prove as satisfactory as the plain disk so was abandoned. Into this illuminator was screwed a small condenser with an iris diaphragm. This made possible a fine control of the amount of light striking the specimen. A slightly greater magnification was obtained by increasing the tube length of the microscope but at no time in the work did the magnification exceed fifty diameters.

(3) Light Source.

At first the ordinary arc light was used but this proved unsatisfactory from the start. The light emitted was too intense and very unsteady. For it was substituted a six volt Mazda lamp operated on a 110 A C circuit by means of a small transformer. This gave a very steady light that never needed attention and one that could always be depended on to be the same. The light from this source was passed through a condenser and then an iris diaphragm and following this another condenser before coming into the microscope. This gave a very satisfactory beam that could be easily and completely controlled.

(4) Instrument used in measuring temperature.

For this work, a rare metal thermocouple was used con-

sisting of Pt and Pt-Rd. It was inserted in the furnace through a hole in the plug holding the leveling device. The end was placed against that part of the device holding the sample and immediately beneath the sample. The ends of the couple were joined to copper leads and the cold junction kept at zero centigrade by immersion in ice in ^aDewar bulb. A Siemens and Halske thermo electric pyrometer graduated in degrees centigrade indicated the temperature.

III EXPERIMENTAL

(1) Procedure in making a run.

The specimen to be observed was ground down to fit the ^Pdepression in the leveling device and then polished and etched. The etching was done with four percent nitric acid in alcohol and the length of time depended on the steel. It was continued until the structure was brought out a sufficient amount to make a clear view under the microscope. The specimen was then placed on the leveling device and this screwed up into the furnace. In adjusting the light, it was necessary to throw it off focus in order to bring out the structure of the specimen. Otherwise the light on being directed down through the two mica windows would strike ^{the} lower one and be partly reflected back on the upper one causing the field to blur. After setting the thermocouple in place and adjusting the microscope, the neutral gas was started through the furnace. Next the cooling agent was started on its path. When sufficient time had passed to allow all the air to be driven out of the furnace, the current was turned on. At first only three or four amperes were used but as the furnace heated up, the amount of current was increased until twelve amperes were reached. This was sufficient to raise the furnace to 900° C. Close observation was kept on the specimen as the heating continued

and any changes were noted. In cooling the specimen, the neutral gas was kept circulating until the furnace was cold in order to prevent any possibility of oxidation.

(2) Different steels tried.

In the selection of a specimen for this work, one was chosen that would show the greatest contrast in structure and thus make it easier to follow any changes that might take place. The first steel tried was a .40% carbon that had been heated to 1000° C and quenched in water, reheated to 625° C and kept there for twenty four hours. This segregated the pearlite and produced a fairly good specimen.

Next a manganese steel was made up of .40% carbon and 7% Mn. This amount of mananese was chosen in order to bring the critical range somewhere between 200° and 300° C. Thus it was hoped to observe the transition stages without the necessity of a very elevated temperature.

The third sample used was a piece of electrolytic iron in the "as cast" condition. This specimen contained pratically no carbon but the crystals were very uniform and rather large.

(3) Different atmospheres tried.

In order to see the specimen at elevated ranges of temperature, it was necessary to exclude any possibility of oxidation. This was done by passing a nonoxidizing gas through the furnace. The first gas tried was carbon dioxide. No results were obtained with it owing to the formation of an oxide film on the specimen probably due to the reduction of some of the CO₂. Next nitrogen was tried. Air was passed through a long tube of hot copper turnings and in this manner fairly pure nitrogn was obtained. After a few runs with this gas it also had to be abandoned because the prod-

uct was not pure enough and the presence of the slightest amount of oxygen was always fatal to the run.

Finally hydrogen was tried and proved the most satisfactory of all. However, even with this powerful reducing agent, it was impossible to prevent the formation of a film over the specimen, causing it to blur.

(4) Rate of heating and cooling.

On starting a run with the furnace, only a small amount of current was used, the amount being increased as the temperature rose. Usually from three to four amperes sufficed at the start. The heating took about fifteen to eighteen minutes. The cooling proceeded very rapidly falling from 800° to 900° C down to about 200° C in approximately three minutes. This usually left the specimen in a sorbitic condition and before another run could be made with the same specimen, it had to be heat treated again, the treatment given depending on the structure desired.

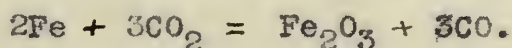
(5) Cooling system used in furnace.

In the first setup of the apparatus, distilled water acted as a cooling agent for the furnace and proved very satisfactory as far as optical qualifications required. However, at higher temperatures, it was rather difficult to get enough water through the furnace to avoid the formation of steam bubbles on the mica window. The steam bubbles forming on the mica possessed a different index of refraction than the water its self and thus caused the light reflected back from the specimen to be rather highly diffused. This prevented seeing the specimen. Later ordinary tap-water was used with the same result. Finally, the practice of water cooling had to be abandoned with this type of furnace.

Air cooling was then substituted and proved satisfactory to a degree. A stream of compressed air was passed in at the top of the furnace and out at the bottom in just the reverse manner in which the water had been circulated. If the furnace was not heated for periods to exceed twenty minutes and a temperature of 900°C this worked very well but if the temperature was raised above this point, the radiation from the top of the furnace endangered the objective of the microscope.

IV RESULTS AND DISCUSSION.

In each of the runs made, the structure could be clearly seen until a temperature of nearly 300°C was reached. In the case of CO_2 and N , the structure became dimmer and dimmer until it passed out altogether. This was between $400 - 425^{\circ}\text{C}$. There was a period of some 100° that nothing could be seen but at about 600°C the structure again became slightly visible. This disappeared completely before 650°C was reached. Similar results were obtained using hydrogen gas but the structure began to disappear at about 500°C reappearing at $550 - 575^{\circ}\text{C}$ and then disappearing completely at 700°C . In the case of CO_2 , the conditions in the furnace were very favorable for the reduction of the gas liberating O_2 which combined with the iron forming an oxide according to the reaction:



The same kind of scale was formed with all the gases. It is probable that traces of oxygen were in the hydrogen gas used. The nitrogen gas was also impure containing varying amounts of oxygen.

V Summary

Although no positive results were obtained, it is be-

lived that this method could be worked out and in the end would prove quite as successful as with the use of a vacuum furnace.

The following suggestions are offered:

1. Absolutely pure and oxygen free hydrogen should be used as the neutral gas.
2. To do away with the light diffraction, transparent quartz should be used in the furnace in place of the mica windows.
3. If possible, the windows should be made larger so that oblique illumination of the specimen could be tried.
4. A swab should be inserted between the windows so that it could be moved across them and thus remove the steam bubbles that collect as the furnace heats.
5. Greater magnification should be used. This can be obtained by mounting another complete microscope in place of the eyepiece of the one microscope (3).

The carrying out of these suggestions will no doubt bring a large measure of success

PART II

THERMAL STUDY OF STEELS WITH
VARYING CARBON CONTENTI INTRODUCTION

This work was undertaken in order to become familiar with the procedure in calibrating thermocouples. Also, to study the thermal changes in a number of steel samples with different carbon content.

II CALIBRATION OF THERMOCOUPLE.

The apparatus used consisted of the following instruments:

1. Hereaas Pt / Pt - Rd couple B&Co No 1.
2. Potentiometer, Leeds and Northrup. direct current standardization apparatus. Model K No. 46246.
3. Galvanometer. Leeds and Northrup. Series Pg. 22893.
4. Standard cell. Weston Standard Cell No. 2939.
5. Storage cell. Lead Storage Cell.
6. Cold Junction. Dewar "Icy-Hot" bottle.
7. Furnace. A small gas fired Mecker Laboratory furnace.

To get the temperature of sulfur vapors a 150 cc hard glass test tube wound with a jacket of rolled asbestos was used.

The point of low calibration was taken as the temperature of vapor from boiling sulfur which was at 444.7°C at 760 mm. As our readings were taken at 746 mm and 24°C they had to be converted.

The point of highest calibration was taken as the freezing point of pure copper under reducing conditions (covered with charcoal). This gave the deflection at a temperature of 1084°C

of 799 m.v. so that $7.99 \text{ m.v.} = 1084^{\circ} \text{C}$ or $1 \text{ m.v.} = 136.9^{\circ} \text{C}$ while for the lower reading $1 \text{ m.v.} = 193.6^{\circ}$. A difference of 56.7° in 640.6° on scale calibration or a correction of $.088^{\circ}\text{C}$ should be added for every degree read. The m.v. values become lower as the temperature rises. This error should be corrected by plotting the curve from the following equation:

$$\text{Log } e = aT + b \quad \text{where } e = \text{m.v.}$$

$T = \text{degrees centigrade.}$

$a + b$ are constants.

From the above data

$$1084^{\circ}\text{C} = 7.99 \text{ m.v.}$$

$$444.3^{\circ}\text{C} = 2.3 \text{ m.v.}$$

$$(1) \text{Log } 2.3/1000 = a \log 444.3 + b$$

$$(2) \text{Log } 7.99/1000 = b \log 1084 + b$$

Solve for b and substitute the values in the equation

$$\text{Log } e = aT + b$$

This correction is so very small that it was neglected and a straight line drawn through the two points.

III PROCEDURE FOR THE CALIBRATION OF THE COUPLE

The metals were melted in a graphite crucible 12 cm deep and about 175 cc capacity. The couple while immersed in the metal was protected by a quartz tube. Before starting, the potentiometer was adjusted against a Weston Standard Cell and the scale deflection of the galvanometer set to read zero. The crucible was then heated to well above the melting point of the metal and allowed to cool. As the cooling occurred, the time was taken on every ten degrees drop in temperature. The freezing point was found by plotting a time temperature curve.

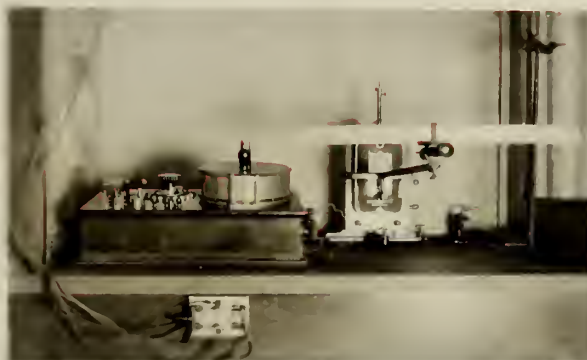


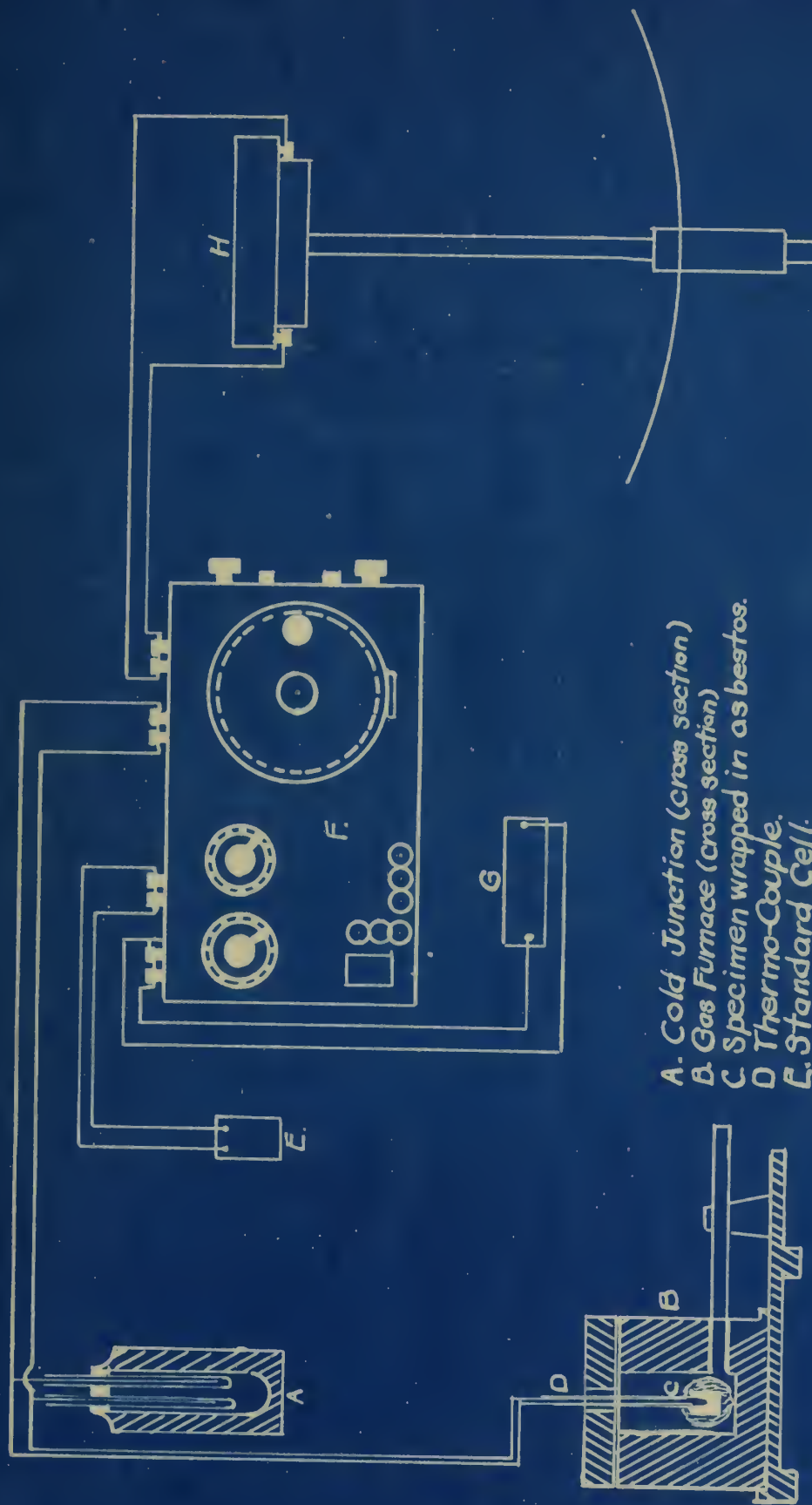
Figure 4.
Potentiometer and Galvanometer.



Figure 5.
Furnace used in heating specimen.

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- A. Cold Junction (cross section)
- B. Gas Furnace (cross section)
- C. Specimen wrapped in asbestos.
- D. Thermo-Couple.
- E. Standard Cell.
- F. Potentiometer.
- G. Storage Battery.
- H. Galvanometer.

Set Up Showing Apparatus Used In Obtaining Cooling Curves

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OF THE
SOCIETY OF ALBANY

Apparatus Used

The apparatus used was the same as that used in calibrating the thermocouple. The potentiometer was the latest make direct reading put out by the Leeds and Northrup Company and has a range from .000001 volt up to 16 volts. It has many desirable features among which may be mentioned, first: There are no contact resistances in the potentiometer circuit proper. Second: it has low internal resistance which gives it the maximum sensitivity in all cases. Compared with high resistance potentiometers, this is especially advantageous in measuring the E.M.F. of thermocouples and the fall of potential across standard low resistances. Third: it is equipped with a double scale which makes it possible to read accurately lower E.M.F.'s than can be read with the older forms of potentiometers.

Cooling curved data was obtained by Osmond's inverse rate method. In this the time required for the specimen to fall successive equal temperature intervals was noted.

When using the specimen, it was wound with several thicknesses of asbestos rope in the hope that the rate of cooling would be uniform. After ^awrapping, the specimen and thermocouple were held suspended in the furnace and heated well above the critical range. The heat was then turned off and the successive intervals of ten degrees fall were carefully timed by using two stop watches. Several runs were made on the same steel sample and the last runs always gave the most uniform results.

IV SPECIMENS USED

Four samples of steel containing .30, .50, .80, and 1.25% carbon were used. These were normalized samples issued by Professor Sauveur in connection with his correspondence course in

metallography.

V. DISCUSSION

The first series of curves obtained were very irregular and their meaning was misleading. This might have been caused by too rapid cooling of the specimen so no asbestos cord was wrapped around the samples and every effort made to retain the heat in the furnace as long as possible. However, the early curves still showed an undue amount of irregularity which, no doubt, was caused by the occluded gas that the specimen contained. This effect has been noted before and studied in some detail by Burgess of the Bureau of Standards, (4) Burgess overcame this difficulty by remelting the samples a number of times. We did not do this as our results after several runs became fairly uniform.

VI CONCLUSIONS

1. The occluded gases should be removed by remelting, preferably in a vacuum, or it will be necessary to take a series of heating and cooling curves at widely different rates in order to determine accurately the critical range.

2. The interval of recording temperature should be wide enough that sufficient sensibility is obtained and narrow enough that the contour of the curves is not distorted.

3. The iron should be in a single piece entirely surrounding and in contact with the thermocouple, otherwise the curves will lose their sharpness due to heat loss. Small samples give sharper curves than large ones

(4) American Institute of Min and Met Eng. 1913 p 2537.

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Part II 1918 page 88

"Changes Within the Critical Range of a Given Steel; From
Ac₁ to Ac_{3.2}"

(2) Zeitschrift für Electrochemie V 15 1909 page 634.
Metallographische Beobachtungen im Luftleeren Raum Bei Hoheren
Temperaturen von P. Oberhoffer.

Abstract: Chem Abstracts 1910 page 160.

Journal of the Society of Chemical Industry
V 28 1909 page 1042.

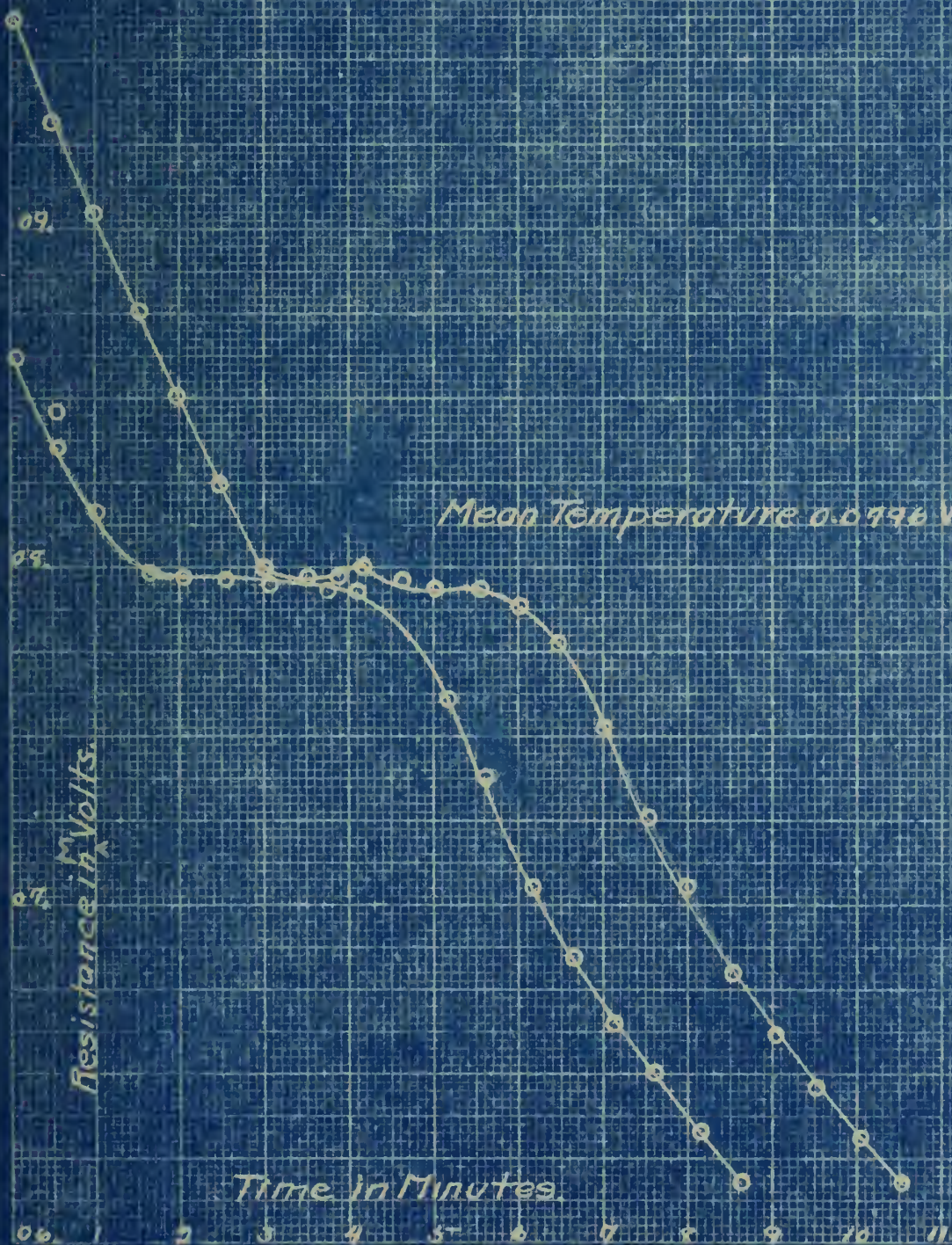
(3) Photomicrography: Eastman Kodak Company page 7.

(4) American Institute of Mining and Met Eng. 1913 page 2537.

Time-Temperature Cooling Curves Showing The Solidification of Pure Copper.

Boiling Point of Sulphur 0.6251 Volts

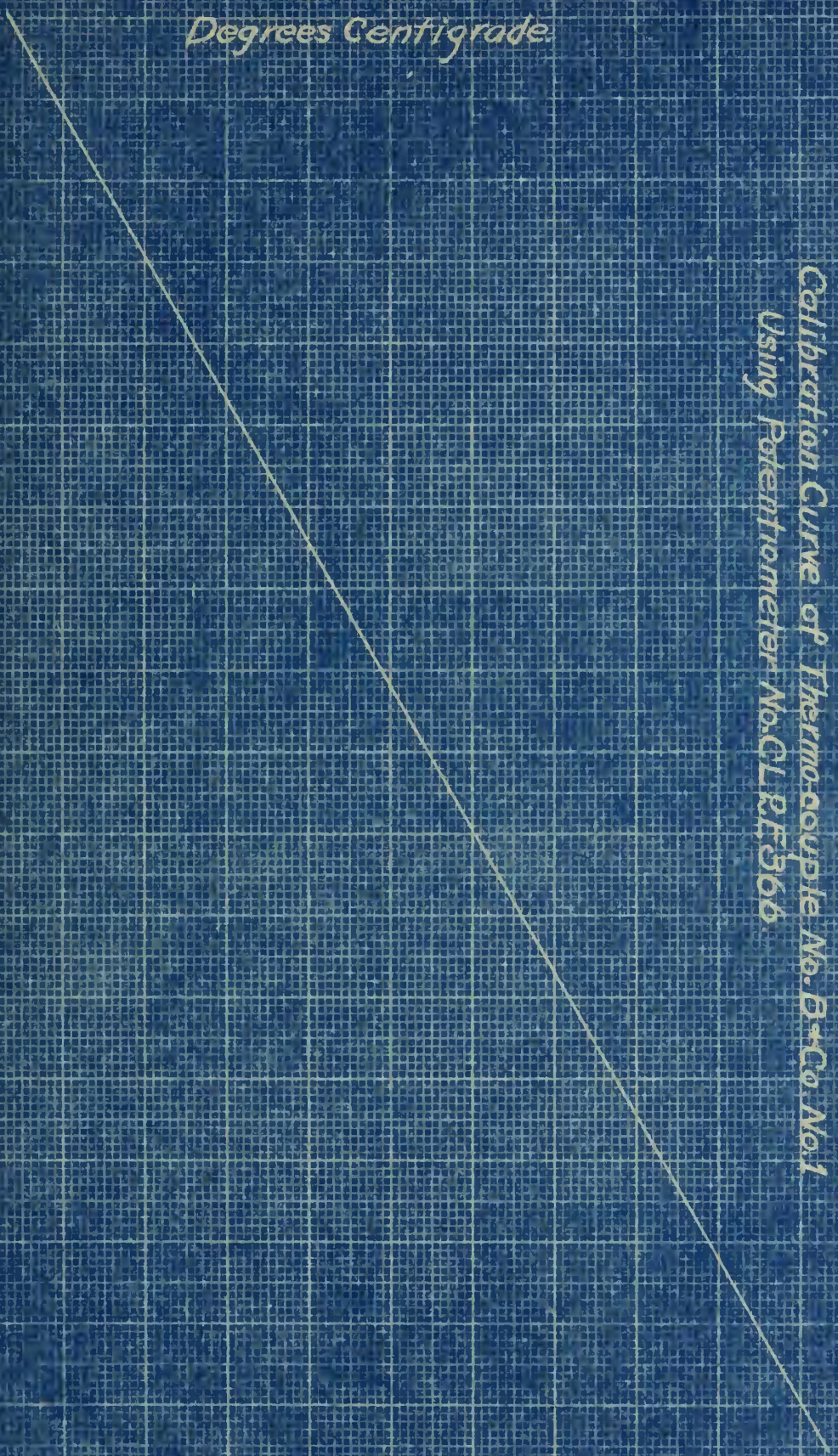
Mean Temperature 0.6796 Volts



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*Calibration Curve of Thermocouple No. B+Co No. 1
Using Potentiometer No. CLRT-566*

Degrees Centigrade.



mV. Volts.

Degrees Centigrade

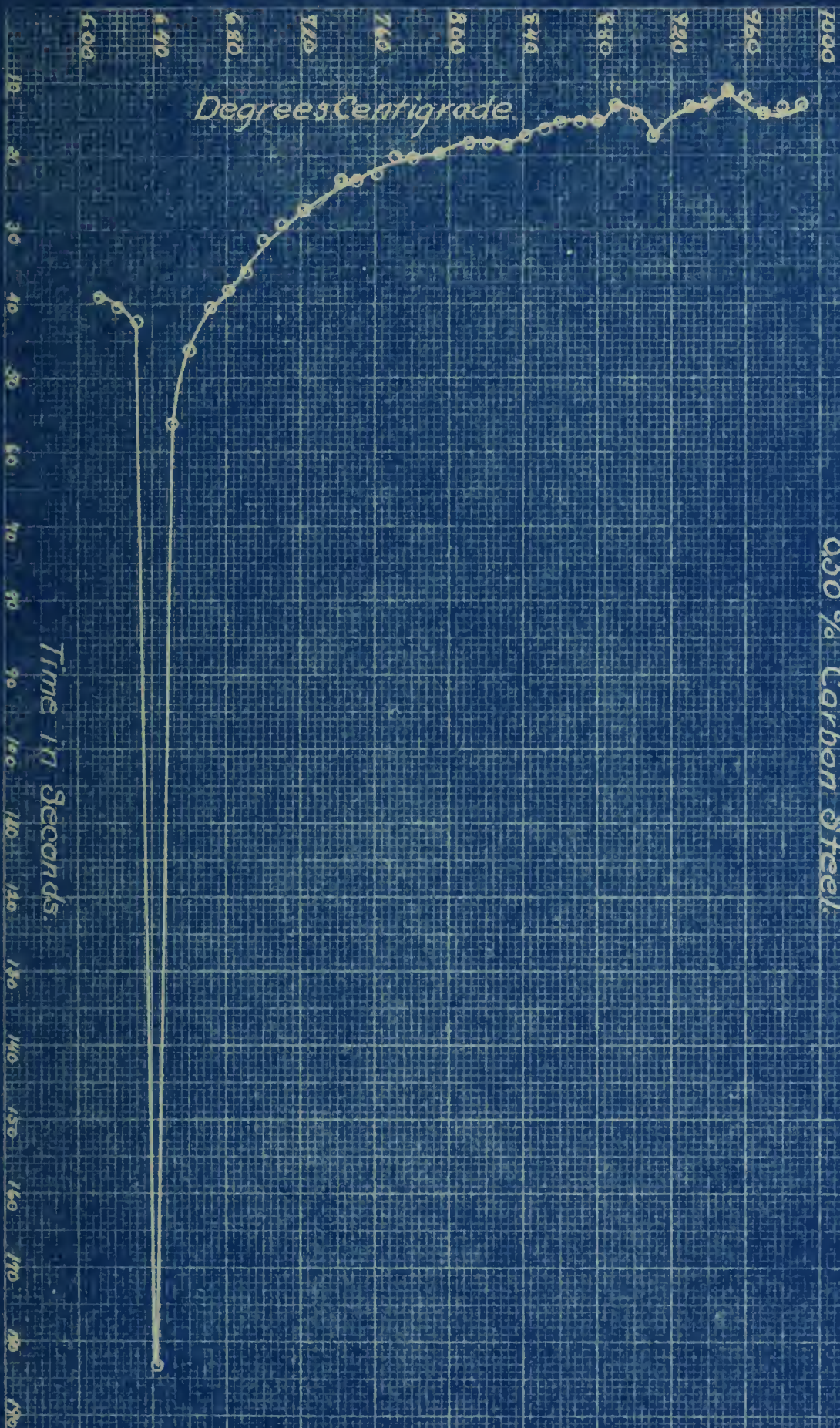
Inverse Rate Cooling Curve
of
0.50 % Carbon Steel

Time in Seconds



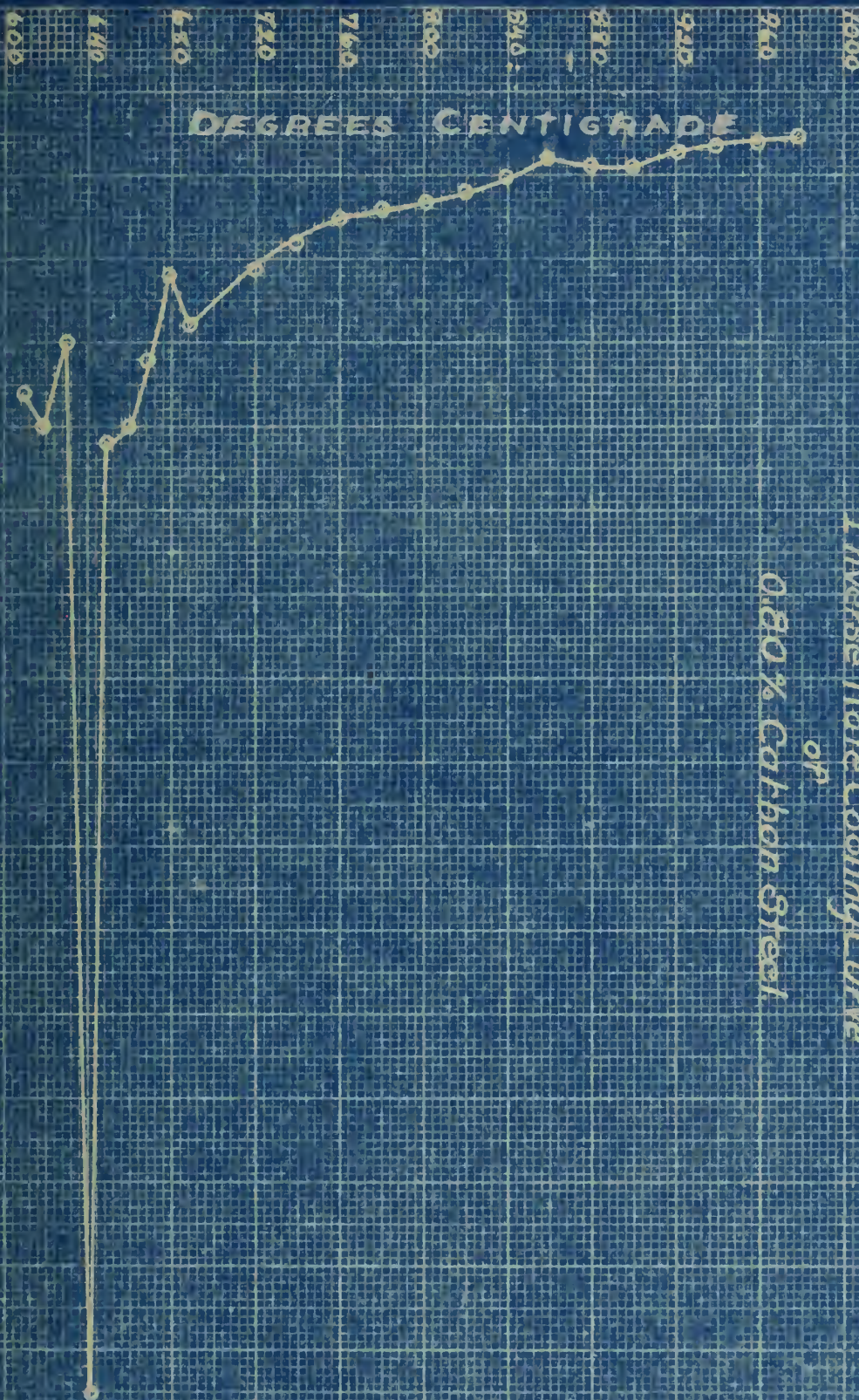
THE HISTORY
OF THE
CITY OF LONDON

Inverse Rate Cooling Curve
of
0.50 % Carbon Steel.



DEGREES CENTIGRADE

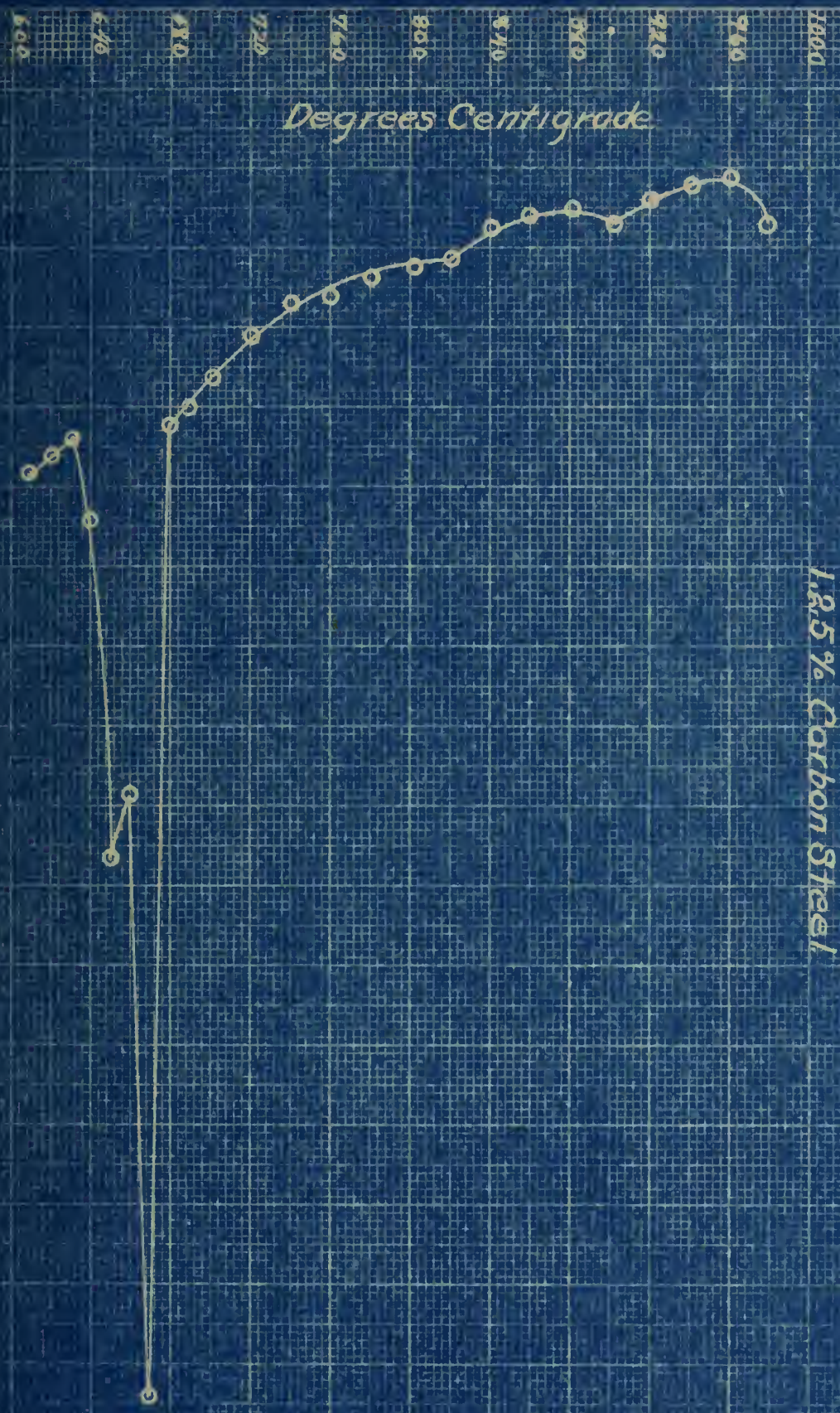
Inverse Rate Cooling Curves
of
0.60% Carbon Steel



Time in Seconds

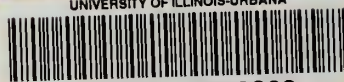
Inverse Rate Cooling Curve of 1.35 % Carbon Steel

Degrees Centigrade



Time in Seconds.

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